L Number	Hits	Search Text	DB	Time stamp
1	1	("5968239").PN.	USPAT; US-PGPUB	2003/04/21 11:18
2	0	(bi adj modal) with slurry with (("SiO.sub.2") or silica)	USPAT; US-PGPUB	2003/04/21 11:19
3	0	(bi adj modal) with slurry with silica	USPAT; US-PGPUB	2003/04/21 11:20
4	5	(bi adj modal) with slurry	USPAT; US-PGPUB	2003/04/21 11:27
5	2	((bi adj modal) with slurry) and silica	USPAT; US-PGPUB	2003/04/21 11:27
6	1	(bi adj modal) with slurry	EPO; JPO; DERWENT; IBM_TDB	2003/04/21 11:24
7	2	(multi adj modal) with slurry	EPO; JPO; DERWENT; IBM_TDB	2003/04/21 11:25
8	2	((multi adj modal) with slurry) not ((bi adj modal) with slurry)	EPO; JPO; DERWENT; IBM_TDB	2003/04/21 11:25
9	0	(((multi adj modal) with slurry) not ((bi adj modal) with slurry)) and silica	EPO; JPO; DERWENT; IBM_TDB	2003/04/21 11:25
10	0	(((multi adj modal) with slurry) not ((bi adj modal) with slurry)) and "SiO.sub.2"	EPO; JPO; DERWENT; IBM_TDB	2003/04/21 11:25
11	2	((bi adj modal) with slurry) and "SiO.sub.2"	USPAT; US-PGPUB	2003/04/21 11:26
12	6	(multi adj modal) with slurry	USPAT; US-PGPUB	2003/04/21 11:27
13	2	((multi adj modal) with slurry) and silica	USPAT; US-PGPUB	2003/04/21 11:27
14	2	(((multi adj modal) with slurry) and silica) not (((bi adj modal) with slurry) and silica)	USPAT; US-PGPUB	2003/04/21 11:27

US-PAT-NO: 6407000

DOCUMENT-IDENTIFIER: US 6407000 B1

TITLE: Method and apparatuses for making and using bi-modal abrasive slurries for mechanical and chemical-mechanical planarization of microelectronic-device substrate assemblies

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A method and apparatus for making and using slurries for planarizing microelectronic-device substrate assemblies in mechanical and/or chemical-mechanical planarization processes. In one aspect of the invention, a **bi-modal slurry** is fabricated by removing a first type of selected abrasive particles from a first abrasive particle solution to form a treated flow of the first solution. The treated flow of the first solution is then combined with a flow of a second solution having a plurality of second abrasive particles. The abrasive particles of the first type are accordingly removed from the first solution separately from the second solution such that the second abrasive particles in the second solution do not affect the removal of the abrasive particles of the first type from the first solution. In another aspect of the invention, a second type of selected abrasive particles are removed from the second solution prior to mixing with the first solution. Thus, by combining the treated flow of the first solution with either the treated or untreated flow of the second solution, a single flow of an abrasive slurry is produced having a first distribution of the first abrasive particles about a first mode and a second distribution of the second abrasive particles about a second mode.

One particularly promising CMP slurry being developed by Micron Technology,

Inc. is a liquid solution having a plurality of first and second abrasive particles. The first and second abrasive particles are typically composed of the same material, such as ceria or **silica** treated ceria abrasive particles. The difference between the first and second abrasive particles is the size of the particles. This **slurry** accordingly has a "bi-modal" distribution of abrasive particles in which the first abrasive particles have particles sizes in a first size distribution about a first mode and the second abrasive particles have particle sizes in a second size distribution about a second mode. In contrast to "singlet" slurries that have only one mode and a signal size distribution of abrasive particles about that mode, bi-modal slurries are expected to exhibit unusually good polishing rates and planarity on both topographical and planar substrate surfaces.

Although bi-modal slurries can produce good results, they may fail to achieve consistent results because the abrasive particles are highly unstable in the solution. The bi-modal slurries mixed by Micron Technology Inc. from components supplied by Rodel Corporation may even change from one planarizing cycle to the next, which greatly increases the difficulty in accurately planarizing substrate assemblies. To resolve the instability of these slurries, a point-of-use filtering may be performed at the planarizing machine of a single flow of a **bi-modal slurry** having both the first and second planarizing particles. Filtering the **bi-modal slurry**, however, may alter the bi-modal distribution of abrasive particles to the extent that the **bi-modal slurry** loses at least some of the advantages of using two different particle sizes. Therefore, there is a need for improved **bi-modal slurry** techniques in CMP processing to achieve the potential advantages of such slurries.

The present invention is directed toward methods and apparatuses for making and using slurries for planarizing microelectronic-device substrate assemblies in mechanical and/or chemical-mechanical planarization processes. In one aspect of the invention, a **bi-modal slurry** is fabricated by removing a first type of selected abrasive particles from a first abrasive particle solution to form a treated flow of the first solution. The treated flow of the first

solution is then combined with a flow of a second solution having a plurality of second abrasive particles. A single flow of an abrasive slurry thus has a first distribution of the first abrasive particles and a second distribution of the second abrasive particles.

In another aspect of the invention, a bi-modal abrasive **slurry** is manufactured by also separating a second type of selected abrasive particles from the second solution prior to combining the first solution with the second solution. Thus, the first and second solutions can be treated independently to avoid affecting the treatment of one solution by treating the other solution.

FIG. 4 is a bar graph illustrating a **slurry** made using a **slurry** manufacturing assembly and method in accordance with one embodiment of the invention having a bi-modal particle size distribution including a first size distribution of first abrasive particles about a first mode and a second size distribution of smaller second abrasive particles about a second mode.

The first solution 212 is a first slurry component of the abrasive slurry 242. The first solution 212 preferably includes water, chemical additives (e.g., dispersants, surfactants, oxidants and other additives), and a plurality of first abrasive particles 216. The first abrasive particles 216 can be aluminum oxide particles, ceria particles, silicon dioxide particles, titanium oxide particles, tantalum oxide particles, ceria treated **silica** particles, or other suitable abrasive particles for removing material from microelectronic device substrate assemblies. The first abrasive particles 216 are preferably the larger particles of a bi-modal abrasive **slurry** having particle sizes from approximately 0.070-1.0 .mu.m, and more preferably from approximately 0.070-0.40 .mu.m. When the first solution 212 is in the first container 210 prior to being mixed with the second solution 222, a significant percentage of the first abrasive particles 216 in the first solution 212 may agglomerate to form first particle agglomerations 218. Each first particle agglomeration 218 may accordingly include two or more individual abrasive particles 216. The individual abrasive particles 216 of the first particle agglomerations 218 are generally bonded together electronically, covalently, or by van der walls

interaction.

The second solution 222 is accordingly a second component of the mixed slurry 242. The second solution 222 generally includes a liquid 224, the same additives that are in the first solution 212, and a plurality of second abrasive particles 226. The second abrasive particles 226 can also be composed of the same material as the first abrasive particles 216 in the first solution 212, such as aluminum oxide particles, ceria particles, silicon dioxide particles, titanium oxide particles, tantalum oxide particles, ceria treated **silica** particles, or other suitable abrasive particles for removing material from microelectronic device substrate assemblies. The second abrasive particles 226 are preferably the smaller particles of a bi-modal abrasive **slurry** having particle sizes from approximately 0.005-0.20 .mu.m. and more preferably from approximately 0.010-0.050 .mu.m. As with the first solution 212, many of the abrasive particles 226 in the second solution 222 may agglomerate into second particle agglomerations 228.

FIG. 4 is a bar graph illustrating a bi-modal particle size distribution of the planarizing **slurry** 242 having a first particle size distribution 280 from approximately 0.20-1.0 .mu.m of the larger first abrasive particles 216 (FIG. 2) and a second particle size distribution 290 from approximately 0.020-0.20 .mu.m of the smaller second abrasive particles 226 (FIG. 2). The first particle size distribution 280 has a first mode 282 identifying that a significant percentage of the first abrasive particles 216 have particle sizes of approximately 0.3-0.4 .mu.m. The second particle size distribution 290 has a second mode 292 identifying that a significant percentage of the second abrasive particles 226 have particle sizes of approximately 0.07-014 .mu.m. In another embodiment (not shown), the first particle size distribution is from approximately 0.070-0.400 .mu.m with a first mode at approximately 0.250-0.300 .mu.m, and the second particle size distribution is from approximately 0.010-0.050 .mu.m with a second mode at approximately 0.020-0.030 .mu.m.

The embodiment of the **slurry** manufacturing assembly 200 and the method of manufacturing the **slurry** 242 described above with reference to FIGS. 2 and 3

are expected to produce bi-modal planarizing slurries with consistent first and second particle size distributions. One aspect of the embodiment of FIGS. 2-4 is the discovery that conventional filtering processes for a **bi-modal slurry** produce inconsistent particle size distributions because the filters remove a disproportionate percentage of the larger first abrasive particles after operating for a period of time. This phenomenon may occur because a common filter sized to remove the upper end of the larger particles is generally too large to also remove agglomerations of the smaller particles. Moreover, as the filter becomes loaded with abrasive particles, the removal rate of larger abrasive particles increases without necessarily increasing the removal rate of the smaller second abrasive particles. The slurry manufacturing system 200 and the methods for making the slurry 242 reduce variations in the first and second particle size distributions because the first and second solutions 212 and 222 are filtered separately to provide more consistent filtering of the individual solutions. The slurry manufacturing system 200 is accordingly expected to have less loading of the filters in a manner that removes a disproportionate percentage of the first abrasive particles 216 from the planarizing solution 242. Thus, the manufacturing system 200 and the methods for manufacturing the planarizing **slurry** 242 are expected to provide more consistent first and second particle size distributions in a **bi-modal slurry**.

The **bi-modal slurry** 242 manufactured in accordance with the method described above with reference to FIGS. 2 and 3 is also expected to produce good planarizing results. Small abrasive particles are expected to planarize highly topographic surfaces much faster than large abrasive particles. Once the surface of the substrate assembly becomes planar, however, slurries with small particles may have a much slower removal rate than slurries with large particles. The bi-modal planarizing solution 242 manufactured in accordance with the embodiment of FIGS. 2-4 includes the small second abrasive particles 226 to provide selective removal of high areas on the substrate surface at an initial stage of a planarizing cycle while the substrate surface has topographical variations. The **bi-modal slurry** 242 also includes the larger first abrasive particles 216 for maintaining a high removal rate once the substrate surface becomes planar. The planarizing solution 242 accordingly

provides selective removal of the topographical features to form a planar surface on the substrate assembly, and then maintains a high removal rate of material from the blanket surface to expediently planarize the substrate assemblies.

L Number	Hits	Search Text	DB	Time stamp
1	0	((triple adj modal) with slurry) and silica	USPAT; US-PGPUB	2003/04/21 14:32
2	0	((triple adj modal) with slurry) and silica	EPO; JPO; DERWENT; IBM_TDB	2003/04/21 14:32
3	0	((multi adj modal) with slurry) and silica	EPO; JPO; DERWENT; IBM_TDB	2003/04/21 14:32
4	6	(multi adj modal) with slurry	USPAT; US-PGPUB	2003/04/21 14:37
5	2	(multi adj modal) with slurry	EPO; JPO; DERWENT; IBM_TDB	2003/04/21 15:09
6	0	(multiple with modal) with slurry	EPO; JPO; DERWENT; IBM_TDB	2003/04/21 15:09
7	12	(multiple with particles) with slurry	EPO; JPO; DERWENT; IBM_TDB	2003/04/21 15:11
8	75	(multiple with particles) with slurry	USPAT; US-PGPUB	2003/04/21 15:17
9	398	(different with particles) with slurry	USPAT; US-PGPUB	2003/04/21 15:29
10	43	((different with particles) with slurry) and cmp	USPAT; US-PGPUB	2003/04/21 15:18
11	87	(different with particles) with slurry	EPO; JPO; DERWENT; IBM_TDB	2003/04/21 15:29
12	3	((different with particles) with slurry) and cmp	EPO; JPO; DERWENT; IBM_TDB	2003/04/21 15:29

US-PAT-NO: 6409781

DOCUMENT-IDENTIFIER: US 6409781 B1

TITLE: Polishing slurries for copper and associated materials

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Further, **different** types of abrasive **particles** were studied to maximize the removal and selectivity characteristic of the **slurry**. Precipitated silica abrasives, with mean size distributions of 4 nm, 8 nm, 13 nm, 20 nm and 70 nm were tested. FIG. 6 shows a TEM picture of 13 nm slurry. The size distribution of these particles is presented in FIG. 7. Fumed silica, with a mean particle size of less than about 700 nm, was also evaluated. All of these mean size distributions can be used to achieve effective polishing rates and selectivities for the first and second slurries.